



Refractory Materials for Flame Deflector Protection System Corrosion Control: Refractory Ceramics Literature Survey

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1 INTRODUCTION AND OBJECTIVE

Ceramics can be defined as a material consisting of hard brittle properties produced from inorganic and nonmetallic minerals made by firing at high temperatures. These materials are compounds between metallic and nonmetallic elements and are either totally ionic, or predominately ionic but having some covalent character. This definition allows for a large range of materials, not all applicable to refractory applications. As this report is focused on potential ceramic materials for high temperature, aggressive exposure applications, the ceramics reviewed as part of this report will focus on refractory ceramics specifically designed and used for these applications.

Ceramic materials consist of a wide variety of products. Callister (2000)¹ characterized ceramic materials into six classifications: glasses, clay products, refractories, cements, abrasives, and advanced ceramics. Figure 1 shows this classification system. This review will focus mainly on refractory ceramics and cements as in general, the other classifications are neither applicable nor economical for use in large structures such as the flame trench. Although much work has been done in advanced ceramics over the past decade or so, these materials are likely cost prohibitive and would have to be fabricated off-site, transported to the NASA facilities, and installed, which make these even less feasible. Although the authors reviewed the literature on advanced ceramic refractories,^{2,3,4,5,6,7,8,9,10,11,12} after the review it was concluded that these materials should not be the focus of this report. A review is in progress on materials and systems for prefabricated refractory ceramic panels, but this review is focusing more on typical refractory materials for prefabricated systems, which could make the system more economically feasible.

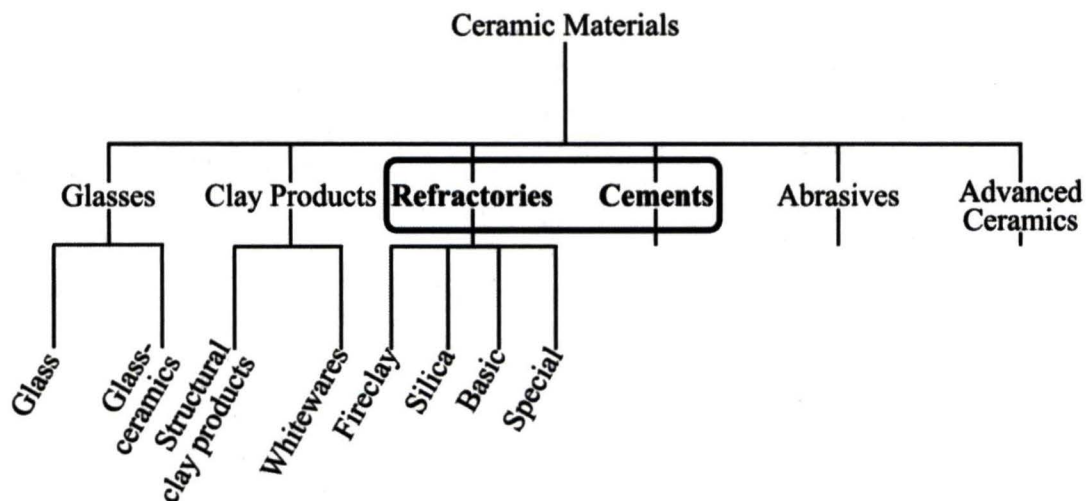


Figure 1. Classification of ceramic materials on the basis of application (after Callister 2000)

Refractory ceramics are used for a wide variety of applications. Figure 2 shows many of these applications, their life expectancy or requirement, and the exposure temperature for the refractory ceramic. Note that the exposure temperatures for refractory ceramics are very similar to the exposure conditions for specialty ceramics (rocket nozzles, space vehicle re-entry fields, etc.) and yet the life expectancy or requirement is relatively low. Currently NASA is repairing

the refractory lining in the flame trench after every launch – although this is not a direct indication of low life expectancy, it does indicate that the current system may not be sufficiently durable to maximize economy. Better performing refractory ceramics are needed to improve the performance, economy, and safety during and after launches at the flame trenches at Kennedy Space Center (KSC).

To achieve this goal a current study is underway to assess different refractory systems for possible use in the flame trenches at KSC. This report will target the potential applicability of refractory ceramics for use in the flame trenches. An overview of the different refractory ceramics will be provided (see Figure 1). This will be followed with a brief description of the structure of refractory products, the properties and characteristics of different systems, the methodology for selecting refractories, and then a general design methodology. Based on these sections, future challenges and opportunities will be identified with the objective of improving the durability, performance, economy, and safety of the launch complex.

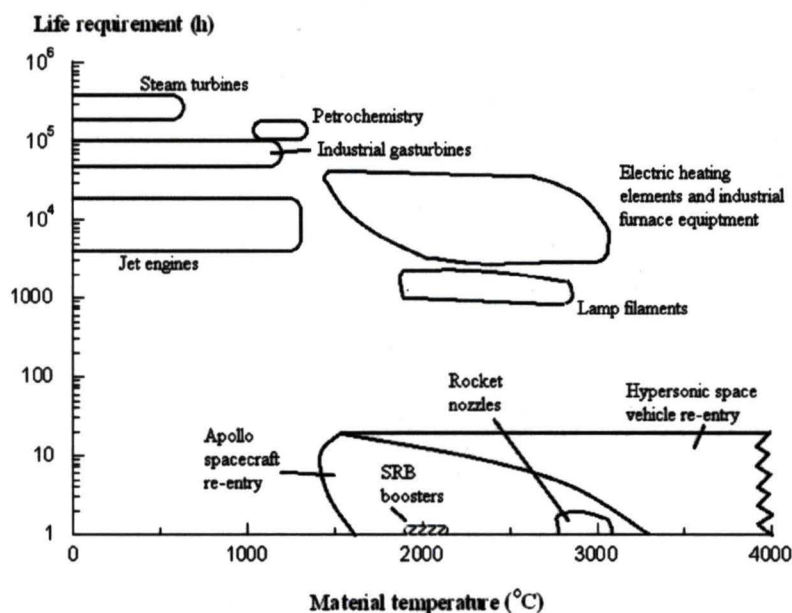


Figure 2. Applications, life requirements, and exposure temperatures for different refractory ceramic applications

2 CERAMIC TYPES

As already noted, there are six general classifications of refractory materials. Each material has unique applications and the material properties must be such that they can endure the exposure for each application. A brief description of each application classification and typical materials used for these applications will be provided.

2.1 Glass

Glass is a familiar group of refractories: containers, windows, and lenses are common products. These materials are non-crystalline silicates containing oxides, typically with other oxides such as CaO, Na₂O, K₂O, or Al₂O₃. The addition of oxide type has a direct influence with the glass' properties. What is unique about glassy materials is that they do not have a specific solidifying temperature – the material increases in viscosity as the temperature is lowered but exhibits no specific melting temperature, T_m . Crystalline materials exhibit a well-defined T_m , with the specific volume exhibiting a significant reduction at this temperature. Glass can be annealed or tempered to achieve a wide variety of properties and characteristics. However, the production process and properties do not meet the requirements for refractory materials in the launch complex.

2.2 Clay-based Products

Clay is one of the most widely used materials for making ceramics. Clay is abundant and often can be used as mined. Most clay-based ceramic products can be sub-classified as structural clays or whitewares. Structural clay products include pipe, tiles, bricks and other products. Whiteware ceramics include porcelain, pottery, tableware, and other similar products. Clay exhibits unique characteristics that make it ideal for ceramic applications: it becomes very plastic when water is added and it melts over a wide range of temperature and can produce relatively dense, strong products with only partial melting (fusing) of the clay. Several fabrication techniques are used to form clay ceramics – these techniques can lead to significant porosity (and lower strengths) of the pre-fired product. Although drying and firing does improve strength, often via vitrification, this method does not produce products with sufficient engineering properties for use in the flame deflector. The fabrication process, that is the firing and drying, also does not lend itself to field installation techniques.

2.3 Refractory Ceramics and Cements

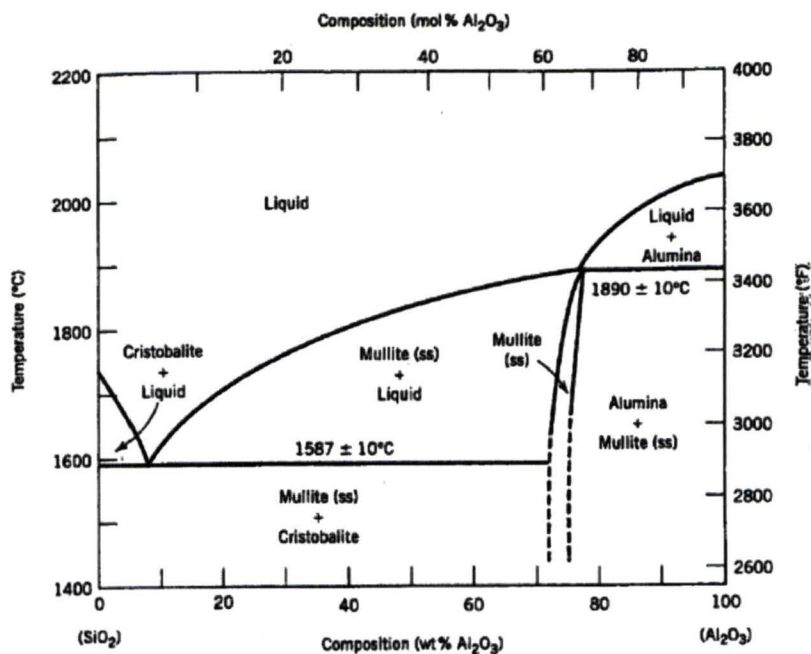
Refractory ceramics and cements can cover a wide range of applications and performance requirements (see Figure 2). Salient properties of refractory materials include the ability to withstand exposure to high temperatures with limited damage and the ability to remain inert when exposed to aggressive environments – both characteristics key for a good flame trench refractory lining. As shown in Figure 1, four general sub-classifications have been defined for refractory products. These sub-classifications typically depend on the use and thus the composition of the refractory material. Table 1 shows typical compositions for common refractory materials.¹³

Table 1. Compositions of Five Common Ceramic Refractory Materials

Refractory Type	Composition (wt%)						
	Al ₂ O ₃	SiO ₂	MgO	Cr ₂ O ₃	Fe ₂ O ₃	CaO	TiO ₂
Fireclay	25-45	70-50	0-1		0-1	0-1	1-2
High-alumina fireclay	90-50	10-45	0-1		0-1	0-1	1-4
Silica	0.2	96.3	0.6			2.2	
Periclase	1.0	3.0	90.0	0.3	3.0	2.5	
Periclase-chrome ore	9.0	5.0	73.0	8.2	2.0	2.2	

2.3.1 Fireclay

The principle ingredients for fireclay refractories are alumina and silica mixtures usually containing between 25 and 45 percent alumina. Figure 3 shows a silica-alumina phase diagram.¹⁴ From this figure it can be seen that for this system the highest temperature possible without melting is approximately 1587 °C. Below 1587 °C the equilibrium phase consists of mullite (3Al₂O₃-2SiO₂) and silica to form cristobalite. In many applications, some melting of the refractory does not result in reduced performance and temperature higher than 1587 °C may be allowed, especially with the short-term exposure to the heated exhaust and the deluge of the flame trenches with water. However, it should be noted that the exhaust of the solid rocket booster (SRB) is approximately 2000 °C, higher than the liquidus line. It should also be noted that the conditions in the launch complex during a launch are far from equilibrium conditions – flame testing could provide valuable information for the non-equilibrium conditions.

**Figure 3. Silica-alumina phase diagram**

The melting point of a fireclay containing 45 percent alumina is approximately 12 percent higher than the melting point of a fireclay with 7.7 percent alumina (eutectic composition) – higher alumina contents for the fireclays will better resist melting.

2.3.2 Silica Refractories

Silica refractories are also known as acid refractories because they resist attack from (or are more compatible with) acidic conditions. In general, compatible phases will not react at elevated temperatures. For the launch exposure conditions, these refractories may be more applicable as they may resist the very acidic conditions from the SRB exhaust. What is important to note is that the addition of alumina, even in small amounts, can reduce the melting point of the material – Figure 3 shows the percent difference in the liquidus line from no alumina to 7.7 percent alumina as being approximately 9 percent.

2.3.3 Basic Refractories

Basic refractories generally contain higher amounts of periclase or magnesia (MgO) but can contain calcium, chromium, and/or iron compounds. The presence of silica in these refractory has a deleterious effect on their high-temperature performance. As the name implies, these refractories tend to perform better when exposed to basic environments. Because the solution that forms from the interactions between the SRB exhaust and the water deluge is very acidic, these refractories may not be suitable for the performance requirements in the flame trench.

2.3.4 Special Refractories

A number of other refractory materials are also available on the market. These include high purity oxides such as alumina, silica, magnesia, beryllia (BeO), zirconia, and carbide compounds, carbon, graphite, and silica-carbide (SiC). However, these high purity oxides and carbon-based materials have a significantly higher cost than the fireclays, silica refractories, and basic refractories. One potential special refractory is geopolymers. Geopolymers are chains or networks of mineral molecules with covalent bonds and are classified as follows¹:

- Waterglass-based geopolymer, poly(siloxonate), soluble silicate, $Si:Al=1:0$
- Kaolinite/Hydrosodalite-based geopolymer, poly(sialate) $Si:Al=1:1$
- Metakaolin MK-750-based geopolymer, poly(sialate-siloxo) $Si:Al=2:1$
- Calcium-based geopolymer, (Ca, K, Na)-sialate, $Si:Al=1, 2, 3$
- Rock-based geopolymer, poly(sialate-multisiloxo) $1 < Si:Al < 5$
- Silica-based geopolymer, sialate link and siloxo link in poly(siloxonate) $Si:Al > 5$
- Fly ash-based geopolymer
- Phosphate-based geopolymer
- Organic-mineral geopolymer

Recent work has shown that geopolymers have good refractoriness – more than conventional concrete but typically less than other refractory products. Research in this area is relatively new but potential opportunities should be further investigated.

¹ <http://www.geopolymer.org/applications/introduction-developments-and-applications-in-geopolymer>; Accessed July 12, 2009.

3 STRUCTURE OF CERAMICS

A thorough discussion on the structure of ceramic materials could (and does) fill significant volumes in the literature – to cover all this is well beyond the scope of this report. However, a general and basic description of the ceramic structure will be presented as the structure effects properties and characteristics. Because ceramics are composed of two or more elements, their crystal structures are much more complex than metallic systems. As already noted, the atomic bonding in these ceramic systems can range from completely ionic to completely covalent, with many containing a mixture of bond types. The degree of ionic bonding, or the ionic character of a ceramic, can be determined as follows:

$$\text{Percent Ionic Character} = \left(1 - e^{(0.25(X_a - X_b)^2)} \right) \cdot 100 \quad (\text{Eq. 1})$$

where X_a and X_b are the electronegativities for the respected elements. Important to note here is that ionic bonds are weaker than covalent bonds. The stability of a ceramic structure is dependent on the sizes or ionic radii of the cations and anions that make up the ceramic. Stable ceramics form when the anions surrounding a cation are in contact with the cation and can be related to the ratio of the ionic radii. This ratio is related to coordination numbers which in turn represent the coordination geometry of the ceramic. These coordination numbers can be related to theoretical densities, which in turn can be related to strength characteristics. A more direct approach to compute the density of a ceramic is to use the following equation:

$$\rho = \frac{n'(\Sigma A_c + \Sigma A_a)}{V_c N_A} \quad (\text{Eq. 2})$$

where n' = the number of formula units in the cell,

ΣA_c = the sum of the atomic weights of all cations in the formula unit,

ΣA_a = the sum of the atomic weights of all anions in the formula unit,

V_c = the unit cell volume, and

N_A = Avogadro's number.

Similar to metallic materials, ceramics will contain imperfections or defects. The structure of common defects includes cation vacancies with a cation interstitial pair (Frenkel defect) and cation vacancy-anion vacancy defects (Schottky defects). Irrespective of the type, these defects can change the characteristics of the ceramic. Impurities can also change the characteristics of ceramics and influence on performance should be determined prior to design and installation.

4 PROPERTIES AND CHARACTERISTICS OF REFRACTORIES

Plain ceramics are brittle materials that exhibit only elastic behavior. Ceramics fail under load by the formation and propagation of microcracks in the materials' microstructure. As the load increases and reaches a critical threshold, these cracks quickly become unstable leading to brittle fracture of the ceramic element. Ceramics are not used where ductile behavior is required. In

most refractory ceramic applications, the design can be accomplished by limiting the tensile forces in the ceramic, thus preventing cracking. However, to design a refractory ceramic system, the properties of the ceramic material are needed.

Testing of ceramic refractories can require different equipment and procedures than conventional concrete materials. Varying testing procedures can result in inaccurate results and incorrect designs. In addition, the thermal history and test temperature can have a significant influence on the mechanical properties and these must be considered during design. Standardized testing of refractory materials is required to assess the characteristics of refractory products and to identify products that can be used in the KSC launch complex. The American Society of Testing and Materials (ASTM) provides standard test procedures for assessing many properties and characteristics of refractory materials – other international societies also provide standards for refractories. The European Committee for Standardization (CEN) has also been very active in producing standards for refractory materials – a list of these is provided in the Appendix.

To properly design ceramic refractory systems, critical material parameters are required. Different environments will require the assessment of different material parameters. The following properties were considered important in an earlier report and include the following:

1. Length change (also referred to as permanent linear change),
2. Cold modulus of elasticity,
3. Hot modulus of elasticity,
4. Thermal conductivity,
5. Cold crushing strength, and
6. Manufacturers' reported operating temperature.

The length change is considered to be an important characteristic as cracking of the ceramic refractory material in the flame deflector can lead to early failure of the refractory, FODS, and accelerated deterioration of the steel base structure.

The modulus of rupture is a critical property in determining the propensity of a ceramic to crack – higher modulus of rupture values will result in less cracking. The exhaust from the launch vehicle imposes loads on the refractory lining, placing the cold face in tension – modulus of rupture testing simulates this condition. When the stress at the bottom cold surface exceeds the extreme fiber stress capacity of the refractory material, the material will crack. Again, higher refractory capacities can take higher demand loads without cracking. Because the temperature conditions at the cold face are unknown, both cold and hot modulus of rupture values should be assessed. Modulus of rupture values for ceramic refractory materials can range from 50 to 1500 psi.

Thermal conductivity is a critical parameter in the design of refractory systems, although for the short duration of heat exposure that occurs during a launch this parameter may be less important than the ceramic material parameters listed above. However, this parameter should be assessed. Thermal conductivity values can range from approximately 10 to 160 W/(m•K).

The cold crushing strength is a commonly reported value and a critical parameter for almost all applications. Standard test methods are available to assess this parameter and it is recommended

that this be assessed as part of a testing program. Cold crushing strengths can range from very low (hundreds of psi) to very high values (to over 10,000 psi).

It is important to evaluate refractory ceramics using test procedures specifically for these materials. Following is a list of ASTM standards for refractory ceramic products:

- Compressive Strength
 - i. ASTM C133-97 (2003) Standard Test Methods for Cold Crushing Strength and Modulus of Rupture of Refractories
- Modulus of Rupture
 - i. ASTM C583-05 Standard Test Method for Modulus of Rupture of Refractory Materials at Elevated Temperatures
- Abrasion/Erosion Resistance
 - i. ASTM C704-07 Standard Test Method for Abrasion Resistance of Refractory Materials at Room Temperature
- Shrinkage and Thermal Expansion
 - i. ASTM C179-04 Standard Test Method for Drying and Firing Linear Change of Refractory Plastic and Ramming Mix Specimens
 - ii. ASTM C832-00 (2005) Standard Test Method of Measuring Thermal Expansion and Creep of Refractories Under Load
 - iii. ASTM C1148-92a (2002) Standard Test Method for Measuring the Drying Shrinkage of Masonry Mortar (Not specifically for refractory concrete — modified)
 - iv. ASTM E228-06 Standard Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer (Not specifically for refractory concrete)

In addition to these properties, the hardness (Vickers or Knoop), fracture toughness, modulus of elasticity, poisson's ratio, and other properties or characteristics may be necessary for proper design – this will depend both on the design procedure and the environment in which the ceramic refractory material is being placed.

5 METHODOLOGY FOR SELECTING REFRACTORIES

The process of selecting a structural ceramic material for use in a particular application and environment involves selecting a material with the best combination of properties and cost for that application and exposure. These properties can include strength, maximum operating temperature, modulus of elasticity, coefficient of thermal expansion, thermal conductivity, and thermal shock resistance. Constructability must also be considered.

The first approach to selecting a refractory material should include the collection and documentation of properties for ceramic materials. Trejo and Zidek reported properties for over 800 refractory products that can be used for this purpose.¹⁶ A preliminary design (discussed in next section) should then be performed to determine general demand values for the environment in which the ceramic refractory material are to be placed. Knowing the demands and potential capacity values of the different properties, an assessment can be performed to maximize the number of capacity values that exceed the demand values.

Ideally, the ratio of capacity to demand values should be high, possibly in the range of 3 to 5. In many applications this is not possible and lower values will have to be used. However, using lower values could lead to earlier failures and higher repair and rehabilitation costs. In some cases section thicknesses may have to be increased or other actions taken to ensure the material capacity is above the load demands. In these cases it is important to consider the additional costs required for these applications.

As with most engineering designs, the process should be iterative – optimizing the design such that a durable, safe system can be constructed at the lowest cost.

6 GENERAL DESIGN METHODOLOGY

Procedures for designing refractory systems have not been standardized. The various applications and exposure conditions that ceramics are exposed make a standardize process difficult and in some cases, incorrect. Considerable care should be taken in cases where safety or economy can be jeopardized.

Ceramics are brittle materials and these should be used as much as possible in compression loading conditions as the compressive load capacity can be more than 10 times the tensile load capacity. Although all attempts should be made to subject the ceramic materials to compressive loads, in almost all cases the actual loading conditions will likely encounter some conditions where tensile loads have to be resisted. An empirical design, or trial and error design, can be performed for all important properties. If demands exceed factored material capacities (in general a reduction factor is applied to actual material properties), another material may have to be selected or the section of the system may have to be changed. What is important is that all characteristics that can affect the performance must be assessed.

The selection of reduction factors should be dependent on the criticality of the property – properties that can lead to significant failures or costs should be lower than lesser important properties. What is important to note is that the designer may not be able to apply the same factor to all parameters and in some cases the demand could be larger than the capacity – however, this should only be allowed to occur in the case when the performance of the system is less dependent on the parameter of interest and failures of this parameter will lead to minor or easily repairable post exposure conditions.

7 CHALLENGES AND OPPORTUNITIES

This report provided a review of ceramic refractory materials with the objective of providing information to the reader such that these materials can be considered for use in the flame trenches at KSC. The existing refractory lining materials are exhibited damage after every launch

and no longer meet the performance requirements for this application and environment – a new system, consisting of a durable, high performing refractory is needed.

The environment conditions in the flame deflector are severe, both during launches and during other times. The environment on the Florida coast is highly corrosive and damage to the refractory lining leads to accelerated corrosion of the steel base structure. Ceramic materials with better properties than the existing materials are available. However, these properties may not be sufficient to resist the damage caused by the exposure environments at KSC. A general overview of ceramics has been provided and this overview can be used to begin the process of identifying critical material parameters and design threshold values – these will identify the requirements for the new refractories for the launch complex.

The challenge is clear – identify or develop a material that can withstand the exposure conditions in the launch complex. The opportunities are improved safety, better performance, improved initial and long-term costs, and reduced resources for the maintenance of the launch complex.

APPENDIX A. CEN/TC 187 – PUBLISHED STANDARDS

Standard reference	Title
CEN/TS 15418:2006	Methods of test for dense refractory products - Guidelines for testing the corrosion of refractories caused by liquids
EN 1094-1:2008	Insulating refractory products - Part 1: Terminology, classification and methods of test for high temperature insulation wool products
EN 1094-2:1998	Insulating refractory products - Part 2: Classification of shaped products (ISO 2245:1990 modified)
EN 1094-4:1995	Insulating refractory products - Part 4: Determination of bulk density and true porosity
EN 1094-6:1998	Insulating refractory products - Part 6: Determination of permanent change in dimensions of shaped products on heating (ISO 2477:1987 modified)
EN 12475-4:1998	Classification of dense shaped refractory products - Part 4: Special products
EN 12698-1:2007	Chemical analysis of nitride bonded silicon carbide refractories - Part 1: Chemical methods
EN 12698-2:2007	Chemical analysis of nitride bonded silicon carbide refractories - Part 2: XRD methods
EN 1402-1:2003	Unshaped refractory products - Part 1: Introduction and classification
EN 1402-2:2003	Unshaped refractory products - Part 2: Sampling for testing
EN 1402-3:2003	Unshaped refractory products - Part 3: Characterization as received
EN 1402-4:2003	Unshaped refractory products - Part 4: Determination of consistency of castables
EN 1402-5:2003	Unshaped refractory products - Part 5: Preparation and treatment of test pieces
EN 1402-6:2003	Unshaped refractory products - Part 6: Measurement of physical properties
EN 1402-7:2003	Unshaped refractory products - Part 7: Tests on pre-formed shapes
EN 1402-8:2003	Unshaped refractory products - Part 8: Determination of complementary properties
EN 14945:2005	Refractory products and materials - Spectrometric determination of chromium (VI) in chrome bearing refractories, before and after use
EN 993-1:1995	Methods of test for dense shaped refractory products - Part 1: Determination of bulk density, apparent porosity and true porosity
EN 993-10:1997	Methods of test for dense shaped refractory products - Part 10: Determination of permanent change in dimensions on heating
EN 993-11:2007	Methods of test for dense shaped refractory products - Part 11: Determination of resistance to thermal shock
EN 993-12:1997	Methods of test for dense shaped refractory products - Part 12: Determination of pyrometric cone equivalent (refractoriness)
EN 993-13:1995	Methods of test for dense shaped refractory products - Part 13: Specification for pyrometric reference cones for laboratory use
EN 993-14:1998	Methods of testing dense shaped refractory products - Part 14: Determination of thermal conductivity by the hot-wire (cross-array) method

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